Halogenated Organic Waste Identification: Qualitative Method

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CHBE 464

January 10, 2014

January 10, 2014

Dr. Christina Gyenge
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Qualitative analysis of halogens in organic waste solutions

Dear Dr. Gyenge,

Enclosed is the final report of the problem-based lab performed from September 26th to October 24th 2013. The scope of the lab was to establish a qualitative method of detecting halogens in chemical waste. In order to reach the project goals, two methods were utilized: the silver nitrate test, and the ammonia test. Silver nitrate was added in the waste samples to check for the formation of solid precipitates, indicating the presence of halogens in the samples. Additional nitric acid solution was added to ensure the solid precipitates formed were silver halides. The silver-nitrate method gave promising results in detecting halogen contaminated containers. Moreover, it was determined that out of the 10 non-halogenated sample containers tested, 6 were contaminated with halogens.

The ammonia test was performed to check the type of halogens present in the waste sample. However, the test proved to be unsuccessful due to various contaminants within the waste samples which made it difficult to distinguish the color-of precipitates formed.

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With just a qualitative method of detecting halogens, determination of the actual halogen concentration in the chemical waste is challenging. A quantitative analysis needs to be performed along with the qualitative analysis performed by this group to ascertain the concentration of halogens in the waste containers.

Sincerely,

Group 2

Encl. Final Report

a place of mind
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AMS SUSTAINABILITY

UBC Chemical and Biological Engineering (CHBE) Problem Based Learning

Halogenated Organic Waste Identification:

Qualitative Method

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Summary

The Environmental Services Facility (ESF) part of the University of British Columbia (UBC) Risk Management Services manages the disposal of hazardous chemical and biological waste generated by the various research laboratories at UBC. In a typical year, the ESF receives over 70 metric tonnes of waste from various university laboratories across the UBC campus of which approximately 17.5% is composed of halogenated organics. Although the UBC Laboratory Pollution Prevention and Hazardous Waste Manual requires each lab to segregate their solvent waste into allocated halogenated and non-halogenated containers, many containers labeled as non-halogenated waste have been found to be contaminated with halogens. Since the disposal method of these two types of waste is quite different, as non-halogenated waste can be recycled while halogenated needs to be incinerated, and due to higher treatment cost for halogenated waste, it is crucial that a simple, reliable and cost-effective protocol be established in order to identify the non-halogenated containers that have been contaminated with halogens.

The ultimate goals of this project are to devise a reliable experimental method to safely identify contaminated waste containers, and to minimize the costs associated with solvent waste disposal. In the first part of the project, we examined the efficiency of a nitric acid and silver nitrate precipitation method in identifying waste containers that were contaminated with halogens. Furthermore two additional set of experiments involving ammonia were tested, in an attempt to identify the type and mass of each halogen present.

The silver nitrate test was proven to be reliable and efficient in identifying 6 out of 10 contaminated containers which were originally labeled as non-halogenated. The dilute and concentrated ammonia tests however were not successful in identifying the type of halogen ion

present in the samples and hence this test will not be continued by the succeeding group of students, who will be continuing on the project.

It should be noted that the funding for this project was provided by the UBC Alma Master Society Sustainability Projects Fund through a Social Ecological Economic Development Studies (SEEDS) grant.

Acknowledgments

We would like to express our very great appreciation to Dr. Christina Gyenge for her valuable and constructive suggestions during the planning and development of this research work. Her willingness to give her time so generously is very much appreciated. We would also like to express our deep gratitude to Ms. Noga Levit and Mr. Bang Dang of the Environmental Service Facility and all other facility members for their patient guidance, and useful critiques towards the resolution of our experiment

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Nomenclature

Term	Description
ESF	Environmental Services Facility
g	Gram
kg	Kilogram
L	Liter
mL	Milliliter
MSDS	Material Safety Data Sheet
ppm	Parts Per Million
rpm	Rotations Per Minute
SEED	Social Ecological Economic Development Studies
SN1	Nucleophilic Substitution
Vol%	Volume Percentage
Wt%	Weight Percentage

1.0 Introduction

The University of British Columbia (UBC) Environmental Services Facility (ESF) receives over 70 metric tonnes of waste from various university laboratories across the campus annually, of which approximately 17.5% is composed of halogenated organics (B. Dang, personal communication, September 2013). The BC threshold limit concentration of halogens in laboratory waste is 1,000 ppm for regular disposal, beyond which off-site incineration is necessary (Hazardous Waste, 2013). Given disposal costs of 1.25 CAD/kg and 1.65 CAD/kg for regular versus halogenated waste respectively, it is of significant economic importance to develop an adequate method of identification and segregation of halogenated from non-halogenated waste.

Figure 1. ESF solvent waste storage

Although the UBC Laboratory Pollution Prevention and Hazardous Waste Manual mandates that halogenated and non-halogenated wastes be segregated into two separate cans (figure 2), these guidelines are not closely adhered to by some UBC laboratories, resulting in cross contamination of non-halogenated with halogenated solvent waste. Given the significant cost associated with off-site testing (~80 CAD/sample) and the large volume of annual waste received by the ESF, it is crucial for an on-site testing protocol to be established to detect contaminations (B. Dang, personal communication, September 2013).

Figure 2. UBC non-halogenated and halogenated solvent waste cans

The general procedure employed in this experiment involves: collection of non-halogenated and halogenated waste containers from the UBC ESF, addition of silver nitrate and nitric acid to each sample, addition of dilute or concentrated ammonia to duplicate samples one and two, respectively, centrifugation of samples, drying of samples, and finally performing a mass balance to determine the quantity of precipitate formed.

This test provides a binary analysis of each sample, in that it either indicates the presence of halogens or lack thereof. A positive test, indicating the presence of halogens, occurs when a precipitate is formed. If no precipitate forms, the waste sample is assumed to contain immaterial halogenated content. The colour of the precipitate indicates which halogen is present, and the ammonia test provides a quantitative basis for mass measurement of each halogen.

This project aims to provide the ESF with a cost effective qualitative experimental method to reliably and safely identify contaminated waste samples. The primary objective of the experiment performed is to examine the efficacy and reliability of a nitric acid and silver nitrate precipitation method in identifying halogenated organics in contaminated samples. The second objective is to identify the type and mass of each halogen present in contaminated samples using ammonia. By implementing this method we aim to minimize the costs associated with solvent waste disposal.

2.0 Theory

The theory behind the silver nitrate test is the SN1 substitution reaction as shown in equation (1) (SN1 Mechanism, 2000). The SN1 mechanism involves two steps: initially a bond of a leaving group is broken and a carbo-cation is formed; in a later step, a nucleophile interacts with the carbo-cation which leads to the formation of the substituted product. Subsequently, dilute nitric acid is added to the solution in order to dissolve any possible confusing halide precipitates that might have formed. Hence, the remaining precipitates will only be silver halides. For instance, silver carbonate is a potential non-halo precipitate that might form in the solution once silver nitrate is added, however upon addition of nitric acid, the carbonate ion will react with nitric acid and hence silver carbonate will dissolve.

$$RX + AgNO_3 \overset{Ethanol}{\qquad} AgX + \ RONO_2 \qquad (1)$$

Since the first step of the SN1 reaction is the time-limiting step, nitric acid is added to the solution 5 minutes after the addition of silver nitrate in order to provide more time for the bond-breaking reaction to occur. Table 1 summarizes the observations of all silver halide precipitates that might form.

The ammonia test reacts silver ions with ammonia to form diamminesilver(I) ion, [Ag(NH3)2]+ as shown in equation (2). This complex is very stable and hence equation (2) lies on the right side of the equilibrium (Clark, 2002). As a result, the silver ion concentration in the solution will decrease. If the multiplication of the new silver ion concentration by the halide ion concentration is less than the solubility product, the precipitate will dissolve according to the reverse equilibrium reaction between silver ions and silver halides.

As can be seen in table 2, only silver chloride precipitates dissolve upon addition of dilute ammonia. If concentrated ammonia is added silver bromide also dissolves. However, silver Iodide precipitates are insoluble in any concentration of ammonia solution. A summary of these observations can be found in table 2.

$$Ag^{+}\ aq\ \ + 2NH_3\ aq \qquad [Ag(NH_3)_2]^{+}(aq) \qquad (2)$$

Table 1 Summary of the observation of silver halides precipitates formed

Ion present in solution	Observation
F^-	no precipitate
Cl^-	white precipitate
Br^-	pale cream precipitate
I^-	pale yellow precipitate

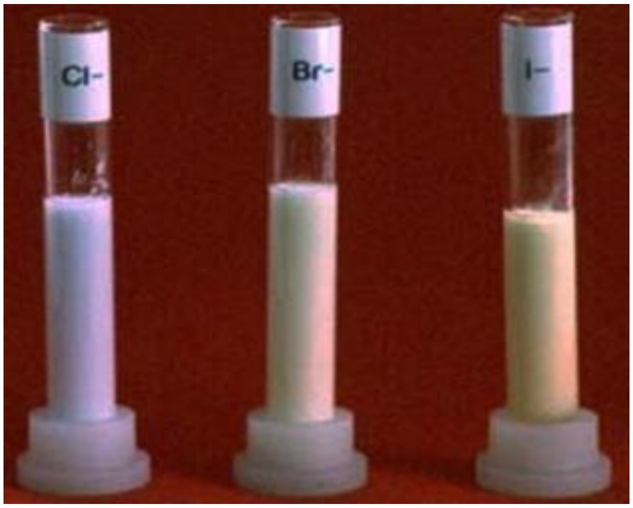

Figure 3. Observation of silver halides precipitates (Clark, 2002)

Table 2 Summary of the observation of the ammonia test (Clark, 2002)

Precipitate	Observation
AgCl	precipitate dissolves to give a colourless solution
AgBr	precipitate is almost unchanged using dilute ammonia solution, but dissolves in concentrated ammonia solution to give a colourless solution
AgI	precipitate is insoluble in ammonia solution of any concentration

3.0 Experimental Apparatus and Techniques

This experiment was conducted in several phases. Raw samples were initially collected at the UBC waste treatment facility. Upon completion of the sampling phase, the samples were carefully packed and delivered to UBC's Chemical and Biological Engineering building under standard protocols for transporting halogenated wastes. Upon arrival, the samples were then processed under a set of carefully planned procedures and experimental conclusions were then deduced. The entire experiment was completed in a six week period during the CHBE 464 problem based learning laboratory.

3.1 Sample Collection

Prior to arrival at the UBC waste treatment facility the following items were procured; twenty disposable 10mL pipette tips, two manual pipettes, twenty 15mL plastic vials and a black felt tip marker. Fifteen waste sample containers were selected and set aside by the waste facility treatment operator.

The entire sample collection process was performed under a fume hood and each individual was required to wear full personal protection equipment (safety glasses, laboratory coat, nitrile gloves, and closed toe shoes) at all times.

To ensure that a uniform homogenous sample was collected, the waste containers were agitated for approximately one minute. A homogenous sample is important as the well-mixed solution is indicative of the entire sample range. Immediately after agitation, the waste container lids were removed and the disposable pipette tips were submerged half way into the container and 5mL of solution was removed and transferred to a 15mL vial. The process was repeated three times until

15mL of solution was collected. The vials were then capped, labelled and safely placed into a polystyrene transfer container. The entire process was further repeated for the remaining fourteen waste containers.

Figure 4. Sampling Collection Area

3.2 Equipment Setup

Before beginning the experiment, initial preparations of chemicals and equipment took place. Sixty empty polyethylene vials were weighed on a digital analytical balance. The mass of the vial and lid were recorded along with the mass of the individual vial. The following labeling system was used to identify each vial:

Table 3 Organized Vial Labeling System

1	2	3	4	5	6	7	8	9	10	11	12	13	14	15
1D	2D	3D	4D	5D	6D	7D	8D	9D	10D	11D	12D	13D	14D	15D
C1	C2	C3	C4	C5	C6	C7	C8	C9	C10	C11	C12	C13	C14	C15
C1D	C2D	C3D	C4D	C5D	C6D	C7D	C8D	C9D	C10D	C11D	C12D	C13D	C14D	C15D

1-15 for solutions with dilute ammonia, 1D-15D for duplicate trials of dilute ammonia, C1-C15 for solutions using concentrated ammonia and C1D-C15D for duplicate trials of concentrated ammonia.

3.3 Chemical Preparation

Concentrated and dilute ammonia were prepared from a stock 5% ammonia solution. Initially, 90mL of water was measured into a 300mL beaker and 10mL of stock ammonia in a 100mL beaker. The ammonia was then decanted into the water containing beaker and diluted to 1000ppm. Dilute ammonia at 100ppm was prepared similarly by mixing 10mL of stock ammonia with 500mL of water. 5wt% silver nitrate was prepared using a magnetic stirrer. Five grams of powder silver nitrate was first weighed and then added to a 300mL beaker containing 100mL of water. A magnetic stir rod was placed into the beaker and the entire solution was agitated on a stir plate for 15 minutes until a clear solution was present. The stock nitric acid solution was supplied as a 5vol% and no dilutions were necessary. 100mL of stock nitric acid was decanted into a 300mL beaker.

3.4 Experimental Proceedings

All experiments were performed under a fume hood, the initial 15mL sample vials were agitated in a vertical motion to ensure a well-mixed solution was present. 5mL sample solutions were then individually pipetted from the 15mL sample vials into the empty labeled 15mL polyethylene vials. A 1mL micropipette was used to facilitate the transfer, this ensured an accurate measurement. Careful disposal and replacement of micropipette tips after every sampling interval ensured that no cross contamination occurred. 1mL of 5wt% silver nitrate and 1mL 5vol% nitric acid were pipetted consecutively into each sample vial and then carefully capped. A gentle agitation in a vertical motion was enforced to ensure that the solutions were properly mixed. Each sample was examined for any precipitation formation. A final 1mL concentrated or dilute ammonia solution was then pipetted into the properly labeled vials and then carefully sealed and agitated once more. Further observations were made and noted for any precipitation formation.

Figure 5. Precipitate formation

Every processed sample was then subjected to centrifugation. Within the centrifuge, all samples were organized in a counter weight fashion so that samples with similar mass were placed directly opposite to one another. The samples were then centrifuged at 4000rpm for 20 minutes.

A simple vacuum pump was constructed in order to remove the supernatant from the compressed pellet. The vacuum pump was constructed using a 1L Buchner flask as the base waste container. A rubber stopper with a one inch outer diameter opening was used to plug the flask top. One inch outer diameter rubber tubing was then threaded through the rubber stopper opening and connected on the opposite end to a glass Pasteur pipette tip. A soft rubber tubing was then attached to the hose barb end of the Buchner flask and then attached on the other end to a polyethylene expansion barbed tee fitting. A larger hosed diameter polyethylene tubing was used to connect the barbed tee to the sink faucet. The remaining opening on the barbed tee was tubed and directed towards the sink. In order to operate the vacuum pump, the water line was opened allowing water to flow in one direction from the faucet across the barbed tee; bypassing the Buchner flask and out into the sink. By bypassing the Buchner flask through the tee, a vacuum is created within the Buchner flask. Performed under a fume hood, the centrifuged sample lids were carefully removed and starting just below the meniscus line, the Pasteur tip was submerged and gradually lowered as the supernatant was removed. Near the pellet, careful vacuuming was considered to ensure that only the supernatant fluid was removed.

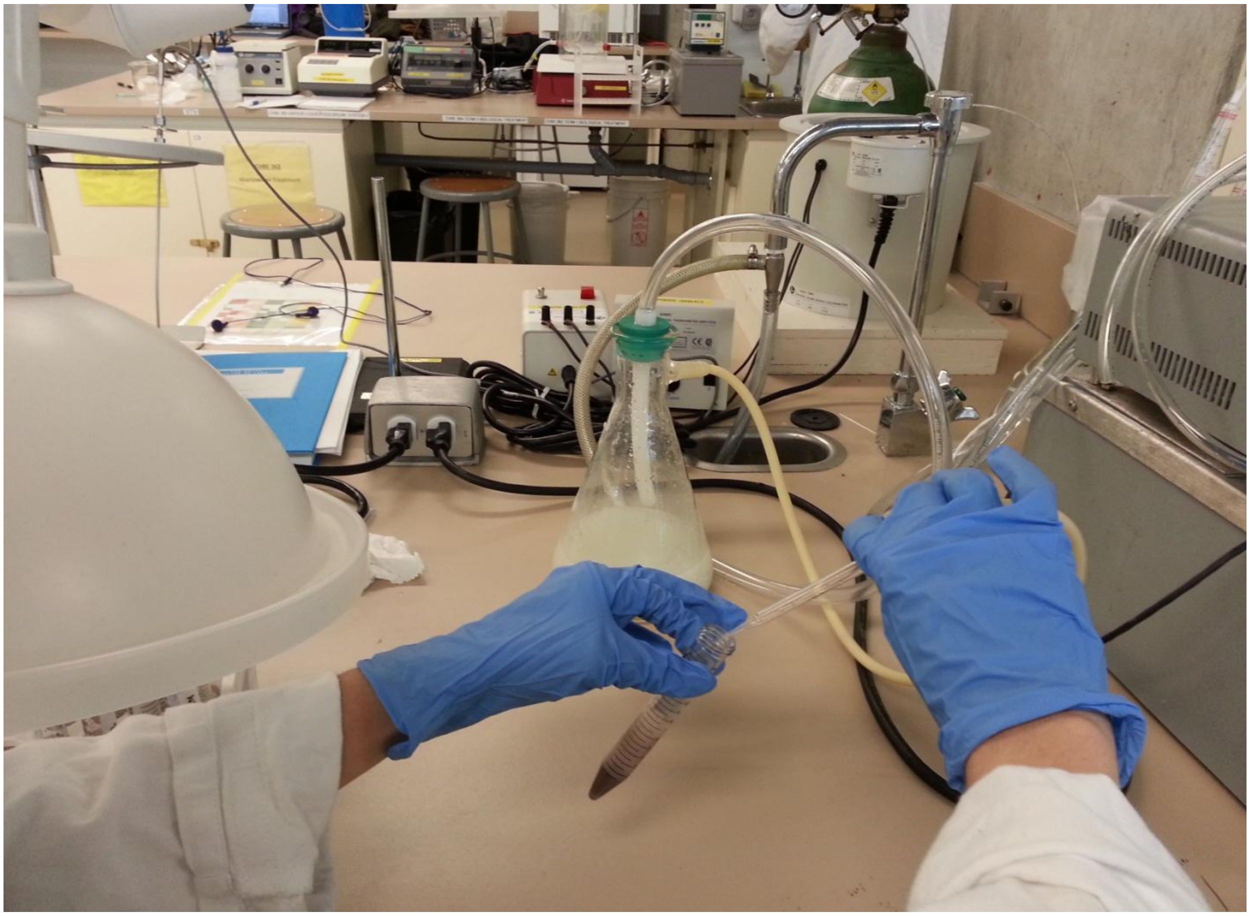

Figure 6. Buchner vacuum suction apparatus

With the removal of the supernatant fluid completed, each vial containing the compressed pellet was then weighed on the analytical balance and then allowed to dry under a fume hood and further weighed again after seven days. The extra drying step was enforced in order to compare the difference in mass between a ‘wet’ and ‘dry’ pellet. The theoretical basis for this step was that the water molecules would evaporate and the difference in mass would be representative of the true quantity of halogens present in the initial sample.

Upon completion of the experiment, all waste liquids were discarded in a waste container labeled ‘halogen wastes’ and all glassware were diluted in water and cleaned.

4.0 Results and Analysis

To achieve the objectives of the project, fifteen 60-mL waste samples were collected from the UBC waste management facility, and three sets of experiments were performed on each sample in two duplicates, resulting in a total number of 90 tests. The first set of tests involved the addition of silver nitrate to the samples with the aim of qualitatively identifying samples that were contaminated with halogens. The second and third tests involved the addition of dilute and concentrated ammonia in order to further confirm presence of halogens in the samples and to identify the types of halogen atoms, i.e. Cl^-, F^- or I^-, present.

The experimental results from these tests will be discussed in detail in the following three sections.

4.1 Qualitative Analysis

In order to identify the contaminated containers, the silver nitrate test (as detailed in the protocol) was performed. Samples #1-10 were obtained from non-halogenated containers while samples #11-15 were taken from halogenated containers. To ensure the accuracy of the test results, all tests were performed on the halogenated samples as well. As can be seen in table 4, waste samples #2-4 and #10 did not form a precipitate, while the rest were found to be contaminated with halogens. In other words, six out of ten containers which were originally marked as non-halogenated were found to be contaminated with halogens. Moreover, the test accurately detected presence of halogens in all 5 containers that were known to be halogenated (#11-15).

As can be found in Table 5 and 6, the dilute and concentrated ammonia tests exhibited the same results: samples #2,3,4 and 10 were found to be non-halogenated. Hence it can be assumed with high certainty that the silver nitrate test is sufficient in qualitative identification of contaminated containers.

4.2 Halogen Identification

In order to qualitatively identify the types of halogens present, three distinctive set of tests were performed: the silver nitrate test and the dilute and concentrated ammonia tests. Silver nitrate and ammonia form coloured precipitates depending on the type of halogens present as shown in Table 4 and 5. As can be seen in Figure 8, the colors of the precipitates formed in these tests were extremely difficult to differentiate especially in the more non-homogeneous multi-layer solutions.

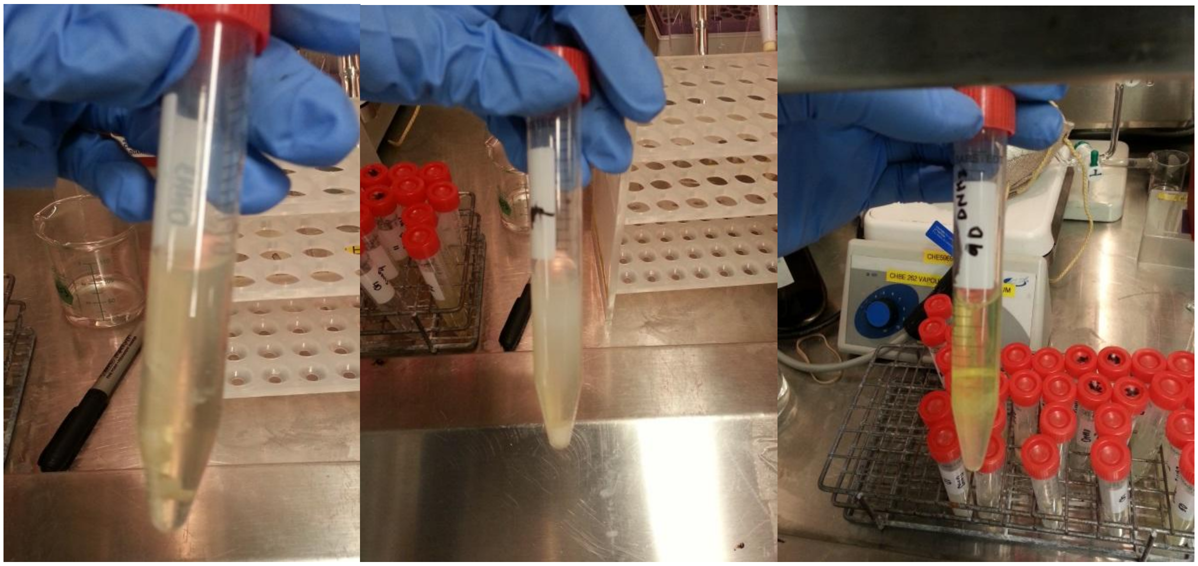

Figure 7. Color of precipitates formed in the ammonia test

4.3 Halogen Quantification

In order to determine the amount of halogen precipitate formed in each test, the samples were centrifuged, dried, and weighed. The net amounts of precipitates were determined by calculating the difference between the mass of each vial with and without solid precipitates. These values can be found in tables 1-3 with m_1 referring to the net mass of precipitate and m_2 the net mass of its associated duplicate sample. Δm denotes the difference between the measured values in the two duplicate samples. As can be seen, the difference between the net amounts of precipitate formed in the two duplicate samples is quite significant in some of the trials, which could be due to a number of facts such as:

- Difficulty in obtaining a homogenized sample from each container that was a good representative of the original
- Removal of some of the suspended solid precipitates in the suction process
- Inaccuracies in the calibration of analytical balance as well as random errors, since on occasions two different readings were obtained for the same sample on two different analytical balances

Table 4. Net Weight of Solid Precipitates Formed with Addition of Silver Nitrate

Sample #	**Halogenated Container**	**Precipitate Formed**	**m_1 (g)**	**m_2 (g)**	**Δm (g)**
1	No	Yes	2.81E-02	2.26E-02	5.50E-03
2	No	No	4.40E-03	1.21E-02	7.70E-03
3	No	No	6.00E-03	4.00E-03	2.00E-03
4	No	No	1.48E-02	7.40E-03	7.40E-03
5	No	Yes	2.07E-01	2.04E-01	3.10E-03
6	No	Yes	1.95E-01	2.09E-01	1.45E-02
7	No	Yes	1.43E-01	1.47E-01	4.10E-03
8	No	Yes	2.08E-01	1.99E-01	9.50E-03
9	No	Yes	4.14E-01	4.45E-01	3.09E-02
10	No	No	1.04E-01	9.40E-03	9.41E-02
11	Yes	Yes	2.37E-01	6.36E-02	1.73E-01
12	Yes	Yes	1.74E-01	1.66E-01	8.00E-03
13	Yes	Yes	9.21E-02	2.73E-02	6.48E-02
14	Yes	Yes	2.55E-01	2.24E-01	3.11E-02
15	Yes	Yes	2.38E-01	1.90E-01	4.75E-02

Table 5. Net Weight of Solid Precipitates Formed with Addition of Silver Nitrate and Dilute Ammonia

Sample #	Halogenated Container	Precipitate Formed	m_1 (g)	m_2 (g)	Δm (g)
1	No	Yes	8.60E-03	1.00E-02	1.40E-03
2	No	No	1.10E-02	1.70E-02	6.00E-03
3	No	No	7.80E-03	2.00E-04	7.60E-03
4	No	No	-3.00E-03	6.00E-04	3.60E-03
5	No	Yes	6.40E-03	7.50E-03	1.10E-03
6	No	Yes	2.87E-02	2.61E-02	2.60E-03
7	No	Yes	2.48E-02	2.14E-02	3.40E-03
8	No	Yes	2.59E-02	2.43E-02	1.60E-03
9	No	Yes	3.13E-02	-1.65E-02	4.78E-02
10	No	No	-6.00E-04	4.00E-04	1.00E-03
11	Yes	Yes	9.10E-03	3.00E-04	8.80E-03
12	Yes	Yes	7.60E-03	7.90E-03	3.00E-04
13	Yes	Yes	1.74E-02	1.50E-02	2.40E-03
14	Yes	Yes	1.90E-02	1.80E-02	1.00E-03
15	Yes	Yes	-1.02E-02	2.00E-02	3.02E-02

Table 6. Net Weight of Solid Precipitates Formed with Addition of Silver Nitrate and Concentrated Ammonia

Sample #	Halogenated Container	Precipitate Formed	m_1 (g)	m_2 (g)	Δm (g)
1	No	Yes	1.84E-02	1.60E-02	2.40E-03
2	No	No	9.20E-03	1.24E-02	3.20E-03
3	No	No	2.70E-03	1.24E-02	9.70E-03
4	No	No	8.70E-03	1.90E-03	6.80E-03
5	No	Yes	1.28E-02	2.78E-02	1.50E-02
6	No	Yes	2.20E-02	2.09E-02	1.10E-03
7	No	Yes	6.23E-02	-5.70E-03	6.80E-02
8	No	Yes	1.14E-02	2.10E-02	9.60E-03
9	No	Yes	1.46E-02	-7.60E-03	2.22E-02
10	No	No	9.10E-03	8.20E-03	9.00E-04
11	Yes	Yes	1.52E-02	1.62E-02	1.00E-03
12	Yes	Yes	4.70E-03	1.32E-02	8.50E-03
13	Yes	Yes	2.10E-03	1.66E-02	1.45E-02
14	Yes	Yes	4.02E-02	1.30E-03	3.89E-02
15	Yes	Yes	1.26E-02	1.22E-02	4.00E-04

5.0 Environmental Considerations

Chlorinated organics pose extreme health and environmental threats (Cleartech, 2010). Halocarbons range from moderately toxic to life threatening with increasing levels of exposure. The primary form of exposure is through inhalation as halogens display a gaseous state at room temperature. Halocarbons are harmful to the human body due to its inability to metabolize the halocarbons rendering an accumulation of halocarbons within the body's fatty tissues (Cleartech, 2010). Health hazards associated with halogenated organics include liver, kidney and respiratory damage, cancer and loss of fertility. Concerning disposal, many halogenated compounds do not degrade in the natural environment and tend to bio-accumulate. Special disposal protocols must therefore be enforced. Many halocarbons are toxic to aquatic organisms in concentrations as low as 137ppm (Cleartech, 2010). Storage is also very hazardous given the potential for evolution of combustible byproducts such as hydrochloric acid and toxic gases such as phosgene gas. Poisonous dioxins and furans may also form, which are powerful carcinogens.

6.0 Safety

Ammonia, ethanol, nitric acid and silver nitrate used in our experiment are considered to be very hazardous (Material Safety Data Sheet Listing). MSDS ratings for all chemicals used pose a health safety rating of three or greater indicating that all four chemicals are very hazardous. Ethanol at high concentrations is extremely flammable and poses a flash temperature of 18 degree Celsius. Due to the toxic and flammable nature of the reagents used, it is recommended that our experiment be conducted under a fume hood and all individuals wear personal protective equipment. Personal protective equipment include: safety gloves, safety goggles, laboratory coat, long pants and closed toe shoes. In the case exposure to the reagents, all contaminated articles of clothing are removed, the exposed flesh is rinsed under cool running water for 15 minutes, use a non-abrasive soap, and then apply a moisturizing lotion. The silver halogenated compounds formed during the experiment are in very low concentrations and although pose a safety threat at high concentrations, should be treated in the same manner as the reagents used if the individual is exposed. The wastes generated from the experiment will be safely disposed by returning the samples back to the ESF.

7.0 Conclusion and Recommendations

This project aims to provide a reliable qualitative testing methodology to systematically identify contaminated non-halogenated samples while minimizing the associated testing costs to allow for adequate segregation and disposal. The objective is to examine the use of silver nitrate on a representative number of samples taken from non-halogenated waste containers in order to determine the reliability and efficiency of this method in detecting halogenated compounds in contaminated samples. The silver-nitrate method that was applied along with the use nitric acid to ensure the presence of halogens in the waste samples gave promising results. Out of the 10 non-halogenated samples and the 5 halogenated samples all 5 halogenated samples and 6 out of the 10 non-halogenated samples revealed that the samples contain halogens inside. The method was relatively simple and short enough to be implied at the UBC Environmental Services Facility for testing.

With just a qualitative method of detecting halogens it is difficult to define the actual halogen concentration in the chemical waste. The halogens can differ from chlorine to fluorine or iodine. The silver nitrate method is not valid for testing the presence of fluorine in the waste sample. Therefore, a quantitative analysis should be performed along with the qualitative analysis performed using the silver nitrate method.

The silver nitrate method applied in the experiment involves the use of nitric acid. Though the concentration of the solution applied for the test is low, it requires the use of concentrated nitric acid while preparation of the solution. Dealing concentrated acid might cause safety issues and should be prevented if possible.

Another method of determining halogens within in organic compounds is the Beilstein method. The method has been used in laboratories and accurate results were produced for detecting the presence of halogens in organic compounds. (Hayman, 1939). The method only uses copper wires to check the presence of halogens in waste samples. The copper wire is burned in open flame, than dipped in the sample and burned in flames once again to check for the existence of halogens in the sample. The presence of halogens will show green flames. However, this method concerns the use of open flames and fumes exerted while burning the sample on the copper wire. However, the method should be given more attention to and be taken into consideration for analysis.

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Appendix A

Raw Data

Table A1: Weight of Solid Precipitates Formed with Addition of Silver Nitrate

Sample #	ID #	Halogenated Container	Precipitate Formed	Mass Empty Vial (1) (g)	Mass Empty Vial (2) (g)	Mass with Solids (1) (g)	Mass with Solids (2) (g)
1	**S0111009073**	No	Yes	6.2085	6.2233	6.2366	6.2459
2	**S081201510**	No	No	6.1042	6.1365	6.1086	6.1486
3	**S081201830**	No	No	6.2717	6.1732	6.2777	6.1772
4	**S081201511**	No	No	6.1935	6.1952	6.2083	6.2026
5	**S081201399**	No	Yes	6.2042	6.1349	6.4114	6.3390
6	**S0111008782**	No	Yes	9.5466	9.4899	9.7412	9.6990
7	**S0111008781**	No	Yes	9.6070	9.5078	9.7500	9.6549
8	**S0111008784**	No	Yes	9.4900	9.5218	9.6981	9.7204
9	**S081201377**	No	Yes	9.6144	9.4746	10.0285	9.9196
10	**S011108670**	No	No	9.5292	6.2304	9.6327	6.2398
11	**S0111000572**	Yes	Yes	6.1899	6.2350	6.4266	6.2986
12	**S081200479**	Yes	Yes	6.2107	6.1509	6.3842	6.3164
13	**S081201081**	Yes	Yes	6.1994	6.1785	6.2915	6.2058
14	**S0309002833**	Yes	Yes	6.1945	6.3106	6.4492	6.5342
15	**S0309002831**	Yes	Yes	6.1741	6.2744	6.4120	6.4648

Table A2: Weight of Solid Precipitates Formed with Addition of Silver Nitrate

Sample #	ID #	Halogenated Container	Precipitate Formed	Mass Empty Vial (1) (g)	Mass Empty Vial (2) (g)	Mass with Solids (1) (g)	Mass with Solids (2) (g)
1	**S0111009073**	No	Yes	6.1955	6.2797	6.2041	6.2897
2	**S081201510**	No	No	6.2078	6.1943	6.2188	6.2113
3	**S081201830**	No	No	6.2004	6.1810	6.2082	6.1812
4	**S081201511**	No	No	6.1614	6.2239	6.1584	6.2245
5	**S081201399**	No	Yes	6.1967	6.2218	6.2031	6.2293
6	**S0111008782**	No	Yes	6.1541	6.1886	6.1828	6.2147
7	**S0111008781**	No	Yes	6.2209	6.1812	6.2457	6.2026
8	**S0111008784**	No	Yes	6.2307	6.2110	6.2566	6.2353
9	**S081201377**	No	Yes	6.1855	6.2104	6.2168	6.1939
10	**S011108670**	No	No	6.2105	6.2003	6.2099	6.2007
11	**S0111000572**	Yes	Yes	6.2015	6.2227	6.2106	6.2230
12	**S081200479**	Yes	Yes	6.1342	6.2109	6.1418	6.2188
13	**S081201081**	Yes	Yes	6.1752	6.2240	6.1926	6.2390
14	**S0309002833**	Yes	Yes	6.2069	6.2169	6.2259	6.2349
15	**S0309002831**	Yes	Yes	6.1631	6.1885	6.1529	6.2085

Table A3: Weight of Solid Precipitates Formed with Addition of Silver Nitrate

Sample #	ID #	Halogenated Container	Precipitate Formed	Mass Empty Vial (1) (g)	Mass Empty Vial (2) (g)	Mass with Solids (1) (g)	Mass with Solids (2) (g)
1	**S0111009073**	No	Yes	6.2280	6.2092	6.2464	6.2252
2	**S081201510**	No	No	6.1810	6.1758	6.1902	6.1882
3	**S081201830**	No	No	6.2265	6.2186	6.2292	6.2310
4	**S081201511**	No	No	6.2013	6.2873	6.2100	6.2892
5	**S081201399**	No	Yes	6.2240	6.1587	6.2368	6.1865
6	**S0111008782**	No	Yes	6.2027	6.2144	6.2247	6.2353
7	**S0111008781**	No	Yes	6.1760	6.1778	6.2383	6.1721
8	**S0111008784**	No	Yes	6.2271	6.2059	6.2385	6.2269
9	**S081201377**	No	Yes	6.2031	6.1412	6.2177	6.1336
10	**S011108670**	No	No	6.2402	6.1439	6.2493	6.1521
11	**S0111000572**	Yes	Yes	6.2167	6.1684	6.2319	6.1846
12	**S081200479**	Yes	Yes	6.1644	6.2098	6.1691	6.2230
13	**S081201081**	Yes	Yes	6.2038	6.1354	6.2059	6.1520
14	**S0309002833**	Yes	Yes	6.1740	6.2036	6.2142	6.2049
15	**S0309002831**	Yes	Yes	6.1737	6.2203	6.1863	6.2325

Appendix B

Sample Calculations

Appendix C

Laboratory Protocol

Laboratory Protocol for the Qualitative Analysis of Haloalkane Compounds in Unknown Contaminated Solutions via Silver Nitrate Test Substitution Reaction

**Safety personal protective equipment must be worn at all times during experimental sessions

1. Sample the unknown solution containing the contaminated halogenated waste.
2. Transfer the sample to a sterilized container.
3. Prepare an ammonia solution at concentrations of 100ppm and 1000ppm.
4. Prepare a 5vol% nitric acid solution and 5wt% silver nitrate solution.
5. Expose the sampled waste solution to 1mL of 100ppm ammonia solution, observe and note for changes.
6. Repeat step 5 on a separate waste sample solution with 1000ppm ammonia solution, observe and note for changes.
7. Pipette 1mL of prepared silver nitrate solution to each test sample, observe and note for changes.
8. Pipette 1mL of prepared nitric acid solution to each test sample, observe and note for changes.